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<p>(21) International Application Number: PCT/EP00/03258 (22) International Filing Date: 11 April 2000 (11.04.00) (30) Priority Data: 99302883.6 14 April 1999 (14.04.99) EP (71) Applicant: SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL). (72) Inventors: HOLMES, Andrew, John; Burleigh Street, Newport, Victoria 3015 (AU). WATSON, Cameron, William; Pool Lane, Ince, Chester, Cheshire CH2 4NU (GB).</p>		<p>(81) Designated States: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report.</i></p>
<p>(54) Title: HYDRAULIC FLUID</p> <p>(57) Abstract</p> <p>A hydraulic fluid comprising a lubricant base oil in combination with (a) from 0.001 to 5 %wt of magnesium salicylate, (b) from 0.01 to 8 %wt of zinc dithiophosphate, and optionally (c) from 0.001 to 5 %wt of a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon atoms; and an additive package comprising magnesium salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon.</p>		

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## HYDRAULIC FLUID

The present invention relates to a hydraulic fluid.

Background of the Invention

WO 93/03121 relates to hydraulic fluids having improved wet filterability and having increased  
5 resistance to loss of zinc when the oil is exposed to water. These compositions comprise (A) a combination of a metal salt selected from the group consisting of sulfonates, phenates, carboxylates and mixtures thereof, (B) an aliphatic carboxylic acid or anhydride, or  
10 carboxylic acid group containing derivative thereof, wherein the aliphatic group contains at least 20 carbon atoms, and optionally (C) a metal salt of (C) (I) at least one organic phosphorus acid or mixture of (C) (I) at least one organic phosphorus acid and (C) (II) at  
15 least one carboxylic acid group, and optionally (D) triazole. In one embodiment, the carboxylic acids of (A) are aromatic carboxylic acids. Zinc, calcium and magnesium salts of these aromatic carboxylic acids, and especially salicylic acids, are preferred. Although  
20 magnesium salicylate and zinc dithiophosphate are both mentioned per se, there is no teaching to use the combination of these compounds.

WO 89/04358 relates to compositions comprising (A) a neutral or basic metal salt of an acidic organic  
25 compound, (B) a metal deactivator, preferably an optionally substituted benzotriazole, and (C) a sulfur and/or phosphorus-containing compound selected from the group consisting of (C-1) phosphorus-containing amide, (C-2) phosphorus-containing ester, (C-3) sulfur-coupled

dithiocarbamate, (C-4) sulfur-containing compound according to formula (I). Particularly preferred metals for the salts (A) are sodium, magnesium, calcium or mixtures of two or more thereof. In the extensive list of additional compounds which can be present, zinc dialkyl dithiophosphate is mentioned. Although magnesium salicylate and zinc dithiophosphate are both mentioned per se, there is no teaching to use the combination of these compounds.

10        EP-A-604 218 describes functional fluids comprising a major amount of an oil of lubricating viscosity, at least one metal salt of thiophosphoric acid, a zinc salt of salicylic acid, and a rust inhibitor, which fluid contains less than about 1 %wt of dispersant and less  
15        than about 1 %wt of basic sulfonate salt. The thiophosphoric acid is most commonly a dihydrocarbyl dithiophosphoric acid; the metal is most preferably zinc. The teaching of the document is restricted to the use of zinc salicylate.

20        In US-A-4,627,928, a process has been described for preparing basic magnesium salicylates which can be characterised as having a magnesium content of at least 150% up to 500%, of the stoichiometrically equivalent amount of magnesium based on the amount of total acid  
25        present. The magnesium salts obtained are described to be suitable for use as additives in general, more specifically as additives for a variety of lubricating oils and fuels, e.g. gasoline and diesel fuels. It is mentioned that these specific magnesium salicylates can  
30        be used in combination with a wide range of other compounds. There is no disclosure or teaching to apply a combination of zinc dithiophosphate and magnesium salicylate in a hydraulic fluid.

Hydraulic fluids need to meet special requirements as they both lubricate and transmit pressure. In order to meet these requirements, hydraulic fluids contain additives. Generally, hydraulic fluids contain  
5 antiwear/extreme pressure additives. The conditions for a hydraulic fluid are especially difficult in vane pumps where the vane tips slide against the casing at high speed under heavy load and at high temperature. Further, the increasingly high operating temperatures of modern  
10 hydraulic equipment requires the fluid to be thermally stable to avoid the formation of deposits and sludge and to resist the corrosion of both ferrous and non-ferrous metals. The requirement of good thermal stability in hydraulic fluids containing an anti-wear additive, means  
15 that these additives are often used in combination with detergents. However, the calcium salts of alkylbenzene sulphonates, alkyl-naphthalene sulphonates, petroleum sulphonates, alkylphenates, alkyl sulphurized phenates or alkylsalicylates which are usually applied, give  
20 increased wear. The increased wear is especially marked in vane pumps, more specifically under low load conditions. Low load conditions occur in practice when the pump is idle.

#### Summary of the Invention

25 It has now surprisingly been found that hydraulic fluid containing a combination of magnesium salicylate and zinc dithiophosphate, gives improved performance at low load compared with a combination containing calcium salicylate. Additionally, it was found that this  
30 combination also provides greater thermal stability. Less sludge and deposits are formed when subjecting the hydraulic fluid of the present invention to the Cincinnati Milacron Thermal Stability Test.

The present invention provides a hydraulic fluid comprising a lubricant base oil in combination with (a) from 0.001 to 5 %wt of magnesium salicylate, (b) from 0.01 to 8 %wt of zinc dithiophosphate, which amounts are based on total weight of hydraulic fluid.

#### Detailed Description of the Invention

The magnesium salicylate for use in the present invention can be either neutral or overbased. The expression "overbased" is equivalent to "basic", "superbased", "hyperbased" and "high-metal containing salts". These magnesium salicylates contain an excess metal content compared to the amount of metal which would be present according to the stoichiometry of the metal and the salicylic acid reacted with the metal. Processes for making such neutral and basic metal salts are well known in the art. Neutral salts can be made by heating a mineral oil solution of an acidic organic compound with a stoichiometric equivalent amount of a metal neutralizing agent such as the metal oxide, hydroxide, carbonate, bicarbonate, or sulfide at a temperature above 50 °C and filtering the resulting mass. Basic salts are made similarly with the exception that a stoichiometric excess of the metal is used.

Preferably, overbased magnesium salicylate is used. These compositions can be characterized by their total base number (TBN). The total base number is preferably at least 100 mg KOH/g, more preferably at least 200 mg KOH/g, most preferably at least 300 mg KOH/g. The total base number is preferably at most 600 mg KOH/g. Another method of characterising overbased magnesium salicylates is by the magnesium content relative to the stoichiometrically equivalent amount of magnesium based on the amount of total acid present. Overbased

magnesium salicyclates for use in the present invention preferably have a magnesium content of more than 500% of the stoichiometrically equivalent amount of magnesium based on the amount of total acid present, more  
5 preferably at least 550%.

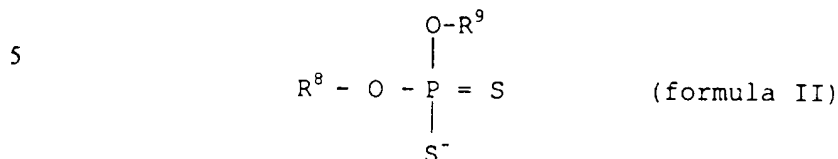
The salicylate can be either substituted or unsubstituted. Suitable substituents include aliphatic groups containing from 1 to 40 carbon atoms and optionally containing one or more oxygen and/or nitrogen  
10 atoms, and hydroxy groups. Preferred substituents are alkyl groups containing from 6 to 30 carbon atoms, preferably from 12 to 20 carbon atoms. Preferably, the substituents are linear. The salicylate can contain from 1 to 4 substituents, preferably from 1 to 3, most  
15 preferably 1 or 2 substituents. Most preferably, the salicylate is substituted by 1 linear alkyl group containing from 14 to 18 carbon atoms.

Generally, mono-alkyl salicylic acids are prepared by alkylation of phenol and subsequent carboxylation.  
20 Therefore, a small amount (generally at most 20 %mol) of dialkyl salicylate and unsubstituted salicylate can be present in the mono-alkyl salicylate.

Magnesium salicylate which can be used in the present invention, is commercially available. A suitable  
25 commercial product is SAP 007 (ex Infineum). The magnesium salicylate will usually be supplied in combination with mineral oil. The amounts referred to in this document, relate to the compound per se without mineral oil.

30 A process by which suitable magnesium salicylate can be prepared, has been described in US-A-4,627,928.

Zinc dithiophosphate is well known in the art. Preferred dithiophosphate for use in the present invention are represented by the following formula



10 in which  $\text{R}^8$  and  $\text{R}^9$  can be either hydrogen or an aliphatic group. The aliphatic group is preferably a hydrocarbyl group. The hydrocarbyl group optionally contains an acid, a hydroxy and/or an ester group. The hydrocarbyl group is preferably an alkyl containing up to 12 carbon atoms optionally further containing an  
15 acid, a hydroxy and/or an ester group. The hydrocarbyl substituted dithiophosphate can contain 1 or 2 aliphatic groups, or it can be a mixture of dithiophosphates having 1 aliphatic group and dithiophosphates having 2 aliphatic groups. Preferably, the zinc dithiophosphate  
20 is a zinc dialkyl dithiophosphate.

Substituted dithiophosphates can contain primary, secondary and/or tertiary aliphatic substituents, i.e. substituents which are attached to the oxygen atom via a carbon atom attached to one, two or three further carbon  
25 atoms, respectively. In the present invention, it is preferred that at least 60 %wt of the aliphatic, preferably alkyl, substituents of the zinc dithiophosphate, are primary aliphatic substituents. More preferably at least 80 %wt of the aliphatic  
30 substituents are primary aliphatic substituents.

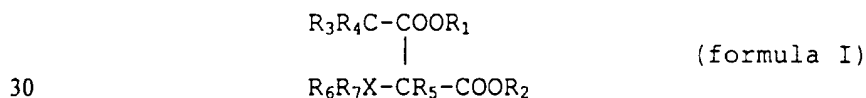
Suitable zinc dithiophosphates which are commercially available include Elco 108 (ex Elco Corporation), Lz 1375 (ex Lubrizol), OLOA 4269Q (ex Oronite) and HITEC 680 (ex Ethyl).



The amount of magnesium salicylate which is present is preferably at most 1 %wt, based on total composition, more preferably at most 0.5 %wt, most preferably at most 0.3 %wt. The amount of magnesium salicylate which is present is preferably at least 0.001 %wt, more preferably at least 0.005 %wt more preferably at least 0.01 %wt, most preferably at least 0.015 %wt. The amount of zinc dithiophosphate which is present is preferably at most 5 %wt, based on total composition, more preferably at most 3 %wt, most preferably at most 1 %wt. The amount of zinc dithiophosphate which is present is preferably at least 0.05 %wt, more preferably at least 0.7 %wt, most preferably at least 0.1 %wt.

The hydraulic fluid can further comprise (c) from 0.001 to 5 %wt of a rust inhibitor based on total composition, preferably a carboxylic acid or ester or amide or imide containing compound, more preferably a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide, more preferably a dicarboxylic acid or its ester, amide or imide containing a total number of between 4 and 70 carbon atoms. More preferably, the number of carbon atoms of the compound is between 20 and 40. A preferred compound is an aliphatic succinimide or succinic diamide. Such compounds have been described in EP-A-776964.

Most preferably, (c) is from 0.001 to 5 %wt of a compound according to the following formula I



in which  $R_1$  and  $R_2$  are each hydrogen or alkyl or hydroxyalkyl of 1 to 30 carbon atoms;  $R_3$ ,  $R_4$  and  $R_5$  are each hydrogen or alkyl or hydroxyalkyl of 1 to 4 carbon

atoms; X is CH or N and R<sub>6</sub> and R<sub>7</sub> are each hydrogen, alkyl or alkenyl of 1 to 30 carbon atoms, or an acyl group derived from a saturated or unsaturated carboxylic acid of up to 30 carbon atoms. Preferably, R<sub>1</sub> and R<sub>2</sub> are  
5 each an alkyl of from 3 to 6 carbon atoms, R<sub>3</sub>, R<sub>4</sub> and R<sub>5</sub> are each hydrogen, X is N and R<sub>6</sub> and R<sub>7</sub> are each an alkyl of 15 to 20 carbon atoms or an acyl group derived from a saturated or unsaturated dicarboxylic acid containing 4 to 10 carbon atoms, at least one of R<sub>6</sub> and  
10 R<sub>7</sub> being an acyl group. Especially preferred is aspartic acid, N-(3-carboxy-1-oxo-2-propenyl)-octadecyl-bis(2-methylpropyl)ester. Such aspartic acid esters are commercially available. Processes for preparing such compounds having been described in EP-A-434 464.

15 The weight ratio in which the compounds of the present invention are present, can differ within wide ranges. Preferably, the hydraulic fluid comprises magnesium salicylate, zinc dithiophosphate and optionally a rust inhibitor in such quantities that the  
20 weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to rust inhibitor is from 1:0 to 1:50.

The lubricant base oils present in the hydraulic  
25 fluid of the present invention, can be any base fluid which is suitable for use in hydraulic fluids. The base oil can be a natural or a synthetic lubricant base oil, or a mixture thereof. The natural oil can be an animal oil or vegetable oil, such as lard oil or castor oil, or  
30 a mineral oil such as liquid petroleum oils and solvent treated or acid treated mineral lubricating oil of the paraffinic, naphthenic, or mixed paraffinic/naphthenic type which may be further refined by hydrocracking and

hydrofinishing processes and/or dewaxing. Synthetic lubricating oils include hydrocarbon oils and halo-substituted hydrocarbon oils such as polymerised and interpolymersed olefins. A suitable base oil contains  
5 poly-alpha-olefins, such as polydecene. Preferably, the base oil is a hydrocarbon base oil. More preferably, the base fluid is a mineral oil which contains less than 10 % by weight of aromatic compounds, preferably less than 5 % by weight, most preferably less than 3.0 % by  
10 weight, measured according to DIN 51378. It is further preferred that the base oil contains less than 1.0 %wt of sulphur, calculated as elemental sulphur, preferably less than 0.1 %wt, more preferably less than 0.05 %wt, measured according to ASTM D 4045. Such mineral oils can  
15 be prepared by severe hydroprocessing. Preferably, the lubricating oil has a kinematic viscosity in the range of from 5 to 220 cSt at 40 °C, more preferably of from 10 to 200 cSt, most preferably of from 20 to 100 cSt.

The hydraulic fluid according to the present  
20 invention can contain further additives usually present in hydraulic fluids, such as pour point depressants, anti-foam agents and demulsifier. Pour point depressants generally are high molecular weight polymers such as alkylaromatic polymers and polymethacrylates. As anti-  
25 foam agents, silicone polymers and/or polymethacrylates are generally used. Demulsifiers which are generally applied are polyalkylene glycol ethers. Furthermore, further detergents such as sulphonates and phenates, metal deactivators, antioxidants such as phenolic  
30 compounds, diphenyl amines and phenyl naphthyl amines, ashless anti-wear agents and/or ashless dispersants, such as succinimides, can be present.

The hydraulic fluids of the present invention contain magnesium salicylate. For specific applications, it can be advantageous that the fluids contain a combination of magnesium and calcium salicylate.

5       The components of the present invention can be added per se to a lubricating oil, or they can be mixed into an additive package before being added to the lubricating oil. The additive package for preparing the hydraulic fluid preferably contains magnesium  
10       salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon atoms, wherein the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5  
15       to 1:100 and the weight ratio of magnesium salicylate to dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide is from 1:0 to 1:50.

#### EXAMPLES

20       Formulations were prepared containing the following additives:

Magnesium salicylate: highly overbased magnesium alkylsalicylate having a total base number of 337 mg KOH/g (SAP 007 ex INFINEUM) and a magnesium content of about 750% of the stoichiometrically equivalent amount  
25       of magnesium based on the amount of total acid, containing 40 %wt of mineral oil  
Calcium salicylate: overbased calcium alkylsalicylate having a total base number of 168 mg KOH/g (SAP 001 ex INFINEUM), containing 40 %wt of mineral oil.

30       The amounts of metal salicylate mentioned in Table 1, are the amounts of metal salicylate per se, without mineral oil.

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Zinc dithiophosphate: zinc di(ethyl-hexyl)dithiophosphate (Elco 108 ex Elco corporation)  
Compound according to formula I: aspartic acid, N-(3-carboxy-1-oxo-2-propenyl)-N-octadecyl , bis(2-methylpropyl)ester.

These compounds were added to a lubricant base oil containing less than 1.0 % by weight of aromatic compounds, less than 0.05 %wt of sulphur, calculated as elemental sulphur and having a kinematic viscosity in the range of 32 cSt (at 40 °) (ISO viscosity grade 32).

The compositions obtained are described in Table 1. The amounts are of the compounds per se, excluding the mineral oil.

Compositions 1 and 2 were used in Vickers V104C vane pump tests. A new pump cartridge was used for each test. The test duration was 250 hours, performed at a fluid temperature of about 66 °C, at a fluid outlet pressure of 35 bar (3.5 MPa) and at a pump speed of 1450 revolutions per minute. The results are described in Table 1.

Table 1

	Composition 1	Composition 2
magnesium salicylate	0.06 %wt	-
calcium salicylate	-	0.06 %wt
25 zinc dithiophosphate	0.37 %wt	0.37 %wt
compound of formula I	0.10 %wt	0.10 %wt
lubricant base oil	balance	balance
Low load test		
ring weight loss (mg)	13	96
30 vane weight loss (mg)	2	1
total weight loss (mg)	15	97

From Table 1 it is clear that better protection against wear is provided by a composition containing magnesium salicylate (1), than by a composition containing calcium salicylate (2). The ring weight loss and total weight  
5 loss incurred when using composition 1 is significantly less than that incurred when composition 2 is employed. The vane weight loss is similar for both compositions.

C L A I M S

1. A hydraulic fluid comprising a lubricant base oil in combination with  
 (a) from 0.001 to 5 %wt of magnesium salicylate,  
 (b) from 0.01 to 8 %wt of zinc dithiophosphate.
- 5 2. A hydraulic fluid according to claim 1, further comprising (c) from 0.001 to 5 %wt of a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon atoms.
- 10 3. A hydraulic fluid according to claim 2, in which (c) is a compound according to the following formula I



- 15 in which  $R_1$  and  $R_2$  are each hydrogen or alkyl or hydroxyalkyl of 1 to 30 carbon atoms;  $R_3$ ,  $R_4$  and  $R_5$  are each hydrogen or alkyl or hydroxyalkyl of 1 to 4 carbon atoms; X is CH or N and  $R_6$  and  $R_7$  are each hydrogen, alkyl or alkenyl of 1 to 30 carbon atoms, or an acyl
- 20 group derived from a saturated or unsaturated carboxylic acid of up to 30 carbon atoms.
4. A hydraulic fluid according to any one of claims 1 to 3, comprising magnesium salicylate, zinc dithiophosphate and optionally a compound according to
- 25 formula I in such quantities that the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to rust inhibitor is from 1:0 to 1:50.

5. A hydraulic fluid according to any one of claims 1 to 4, which fluid further comprises pour point depressant, anti-foam agent and/or demulsifier.

6. Additive package for preparing a hydraulic fluid  
5 which additive package comprises magnesium salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon,  
10 wherein the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide is from 1:0 to 1:50.



## INTERNATIONAL SEARCH REPORT

Intern. Application No

PCT/EP 00/03258

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10M163/00 C10M141/10 //(C10M163/00,133:06,137:10,159:20),  
(C10M141/10,129:54,133:06,137:10),C10N10:04,40:08

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EPO-Internal, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4 462 918 A (MATTHEWS PETER H D ET AL) 31 July 1984 (1984-07-31) the whole document ---	1-6
Y	US 4 627 928 A (KARN JACK L) 9 December 1986 (1986-12-09) cited in the application column 18, line 5-8; examples A,B ---	1-6
Y	EP 0 552 892 A (ETHYL PETROLEUM ADDITIVES INC) 28 July 1993 (1993-07-28) page 3, line 27-34; examples 1-4; tables 1,2 page 9, line 12-29; claim 1 --- -/--	1-6

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

26 July 2000

Date of mailing of the international search report

03/08/2000

Name and mailing address of the ISA

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## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 00/03258

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 92 19703 A (LUBRIZOL CORP) 12 November 1992 (1992-11-12) page 1, line 13-31 abstract ----	1-6
X	WO 97 10318 A (EXXON CHEMICAL PATENTS INC) 20 March 1997 (1997-03-20) page 20, line 15-25 page 24, line 21 -page 25, line 22; examples 1,2; tables I,II ----	1,2,4-6
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